# Effects of air-abrasion pressure on the resin bond strength to zirconia: a combined cyclic loading and thermocycling aging study

Al-Shehri, Eman Z ; Al-Zain, Afnan O ; Sabrah, Alaa H ; Al-Angari, Sarah S ; Al Dehailan, Laila ; Eckert, George J ; Özcan, Mutlu ; Platt, Jeffrey A ; Bottino, Marco C

Abstract: OBJECTIVES To determine the combined effect of fatigue cyclic loading and thermocycling (CLTC) on the shear bond strength (SBS) of a resin cement to zirconia surfaces that were previously air-abraded with aluminum oxide ($Al_2O_3$) particles at different pressures. MATERIALS AND METHODS Seventy-two cuboid zirconia specimens were prepared and randomly assigned to 3 groups according to the air-abrasion pressures (1, 2, and 2.8 bar), and each group was further divided into 2 groups depending on aging parameters (n = 12). Panavia F 2.0 was placed on pre-conditioned zirconia surfaces, and SBS testing was performed either after 24 hours or 10,000 fatigue cycles (cyclic loading) and 5,000 thermocycles. Non-contact profilometry was used to measure surface roughness. Failure modes were evaluated under optical and scanning electron microscopy. The data were analyzed using 2-way analysis of variance and $\chi^2$ tests ($\alpha = 0.05$). RESULTS The 2.8 bar group showed significantly higher surface roughness compared to the 1 bar group ($p < 0.05$). The interaction between pressure and time/cycling was not significant on SBS, and pressure did not have a significant effect either. SBS was significantly higher ($p = 0.006$) for 24 hours storage compared to CLTC. The 2 bar-CLTC group presented significantly higher percentage of pre-test failure during fatigue compared to the other groups. Mixed-failure mode was more frequent than adhesive failure. CONCLUSIONS CLTC significantly decreased the SBS values regardless of the air-abrasion pressure used.

DOI: https://doi.org/10.5395/rde.2017.42.3.206



Originally published at:
Al-Shehri, Eman Z; Al-Zain, Afnan O; Sabrah, Alaa H; Al-Angari, Sarah S; Al Dehailan, Laila; Eckert, George J; Özcan, Mutlu; Platt, Jeffrey A; Bottino, Marco C (2017). Effects of air-abrasion pressure on the resin bond strength to zirconia: a combined cyclic loading and thermocycling aging study. Restorative Dentistry Endodontics, 42(3):206-215.
DOI: https://doi.org/10.5395/rde.2017.42.3.206

Restor Dent Endod. 2017 Aug;42(3):206-215
https://doi.org/10.5395/rde.2017.42.3.206
pISSN 2234-7658·eISSN 2234-7666

Restorative Dentistry & Endodontics RDE

Research Article

Check for updates

# Effects of air-abrasion pressure on the resin bond strength to zirconia: a combined cyclic loading and thermocycling aging study

**Eman Z. Al-Shehri,[1,2,†] Afnan O. Al-Zain,[1,3,†] Alaa H. Sabrah,[1,4] Sarah S. Al-Angari,[1,2] Laila Al Dehailan,[1,5] George J. Eckert ,[6] Mutlu Özcan,[7] Jeffrey A. Platt,[1] Marco C. Bottino [1*]**

[1]Division of Dental Biomaterials, Department of Biomedical and Applied Sciences, Indiana University School of Dentistry, Indianapolis, IN, USA
[2]Department of Restorative Dental Sciences, King Saud University College of Dentistry, Riyadh, Kingdom of Saudi Arabia
[3]Department of Operative Dentistry, King Abdulaziz University Faculty of Dentistry, Jeddah, Kingdom of Saudi Arabia
[4]Department of Conservative Dentistry, The University of Jordan College of Dentistry, Amman, Jordan
[5]Department of Restorative Dental Sciences, University of Dammam College of Dentistry, Dammam, Kingdom of Saudi Arabia
[6]Department of Biostatistics, Indiana University School of Medicine, Indianapolis, IN, USA
[7]Dental Materials Unit, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, University of Zurich, Zurich, Switzerland



**Received:** Feb 25, 2016
**Accepted:** Apr 6, 2017

Al-Shehri EZ, Al-Zain AO, Sabrah AH, Al-Angari SS, Al Dehailan L, Eckert GJ, Özcan M, Platt JA, Bottino MC

***Correspondence to**
**Marco C. Bottino, DDS, MSc, PhD**
Associate Professor, Division of Dental Biomaterials, Department of Biomedical and Applied Sciences, Indiana University School of Dentistry, 1121 W. Michigan Street, Rm. DS270, Indianapolis, IN 46202, USA.
Tel: +1-317-274-3725
Fax: +1-317-278-7462
E-mail: mbottino@iu.edu

[†]Eman Z. Al-Shehri and Afnan O. Al-Zain contributed equally to this work.



**Conflict of Interest**
No potential conflict of interest relevant to this article was reported.

**Author Contributions**
Conceptualization: Bottino MC; Data curation: Sabrah AH, Al-Zain AO, Bottino MC; Formal analysis: Eckert GJ; Funding acquisition:

## ABSTRACT

**Objectives:** To determine the combined effect of fatigue cyclic loading and thermocycling (CLTC) on the shear bond strength (SBS) of a resin cement to zirconia surfaces that were previously air-abraded with aluminum oxide ($Al_2O_3$) particles at different pressures.
**Materials and Methods:** Seventy-two cuboid zirconia specimens were prepared and randomly assigned to 3 groups according to the air-abrasion pressures (1, 2, and 2.8 bar), and each group was further divided into 2 groups depending on aging parameters ($n = 12$). Panavia F 2.0 was placed on pre-conditioned zirconia surfaces, and SBS testing was performed either after 24 hours or 10,000 fatigue cycles (cyclic loading) and 5,000 thermocycles. Non-contact profilometry was used to measure surface roughness. Failure modes were evaluated under optical and scanning electron microscopy. The data were analyzed using 2-way analysis of variance and $\chi^2$ tests ($\alpha = 0.05$).
**Results:** The 2.8 bar group showed significantly higher surface roughness compared to the 1 bar group ($p < 0.05$). The interaction between pressure and time/cycling was not significant on SBS, and pressure did not have a significant effect either. SBS was significantly higher ($p$ = 0.006) for 24 hours storage compared to CLTC. The 2 bar-CLTC group presented significantly higher percentage of pre-test failure during fatigue compared to the other groups. Mixed-failure mode was more frequent than adhesive failure.
**Conclusions:** CLTC significantly decreased the SBS values regardless of the air-abrasion pressure used.

**Keywords:** Air-abrasion; Bond strength; Fatigue; Panavia F 2.0; Resin cement; Thermocycling

Bottino MC; Investigation: Al-Shehri EZ, Al-Zain AO, Sabrah AH, Al-Angari SS, Al Dehailan L; Methodology: Bottino MC; Project administration: Bottino MC, Al-Zain AO; Resources: Bottino MC; Supervision: Bottino MC; Validation: Bottino MC, Platt JA; Visualization: Al-Zain AO, Sabrah AH; Writing - original draft: Al-Shehri EZ, Al-Zain AO, Sabrah AH, Al-Angari SS, Al Dehailan L; Writing - review & editing: Al-Shehri EZ, Al-Zain AO, Bottino MC, Platt JA, Özcan M.

**ORCID iDs**

George J. Eckert
https://orcid.org/0000-0001-7798-7155
Marco C. Bottino
https://orcid.org/0000-0001-8740-2464

## INTRODUCTION

Yttrium oxide-stabilized tetragonal zirconia polycrystal (Y-TZP) is frequently used in dentistry due to its outstanding mechanical properties, biocompatibility, and aesthetic performance [1-4]. These superior properties made zirconium dioxide ceramics a popular high-strength ceramic with a large variety of clinical applications [1-5]. However, its chemical inertness challenges establishment of a strong, durable bond with other materials [5-8]. The composition and physical properties of zirconia ceramics differ substantially from silica-based ceramics, and require alternative bonding techniques to achieve strong and durable bonding of resin [7,8].

The clinical success of resin bonding procedures for ceramic restorations depends on the quality and durability of the bond between ceramic and resin cements. The quality of the bond depends on several factors, such as the bonding mechanisms that are controlled by the surface treatment, which promotes micromechanical and/or chemical bonding to ceramics [8]. Mechanical retention of adhesives to zirconia ceramics can be achieved by air-abrasion or tribochemical silica coating before using chemical bonding agents as organosilanes or ceramic primers. The aforementioned chemical agents promote better interaction with the ceramic surface by increasing the surface energy, and in turn, the wettability of the cement [9-14]. Dentin adhesives containing an organophosphate ester monomer, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP), 4-methacryloxyethyl trimellitate anhydride (4-META), 6-methacryloyloxyhexyl phosphonoacetate (6-MHPA), or 6-methacryl-oxyhexyl 2-thiouracil-5-carboxylate (MTU-6), were shown to activate zirconia surfaces [14]. Consequently, cements containing these monomers have led to higher bond strength when used with zirconia, and even higher bond strength when combined with air-abraded zirconia [14-17]. However, there is no consensus in the literature regarding the effective air-abrasion procedure to improve the resin cement adhesion to the zirconia ceramics [18-21].

Thermocycling has been widely used to simulate thermal stresses commonly occurring in the oral environment based on differences in the coefficient of thermal expansion of materials [22]. However, thermocycling alone does not precisely mimic oral conditions. Adding fatigue cyclic loading may provide better assessment of the clinical performance of adhesive systems [23]. Recent meta-analysis studies reported on different protocols that involved subjecting test specimens to either thermal stresses or mechanical fatigue in an occlusal direction or perpendicular to the adhesive interface, but not a combination of these methods in a shear direction [15,18]. One study evaluated the effect of fatigue cycling (*i.e.*, 26 N at 2 Hz for 27,500 cycles) on the shear bond strength (SBS) of a resin/porcelain system [24]. Considering the failure mode that occurs in the oral cavity, the proposed *in vitro* testing method may provide more clinically relevant evaluation of bond strength between zirconia and adhesive cements.

To the best of our knowledge, the effects of different air-abrasion pressures on surface roughness and adhesion performance to zirconia ceramic after combining thermal aging and mechanical fatigue cyclic loading in a shear direction have not been investigated. Therefore, the overall goal of this study was to determine the combined effect of fatigue cyclic loading and thermocycling (CLTC) on the SBS of a resin cement to Y-TZP zirconia surfaces prepared at different air-abrasion pressures. The null hypotheses tested were: 1) increasing air-abrasion pressure using aluminum oxide ($Al_2O_3$) particles would not affect Y-TZP zirconia ceramic surface roughness; and 2) the combined effect of fatigue CLTC would not affect the SBS of resin cement to Y-TZP zirconia surfaces prepared at different air-abrasion pressures.

 https://doi.org/10.5395/rde.2017.42.3.206 

## MATERIALS AND METHODS

### Y-TZP surface treatment

Seventy-two cuboid samples (10 × 10 × 2 mm) were sectioned before sintering from a disk-shaped block of Y-TZP zirconia (Ivoclar Vivadent Inc., Amherst, NY, USA) using Isomet 1000 (Buehler, Lake Bluff, IL, USA). Specimens were dried in an oven (Cerampress QEX porcelain and processing furnace, Dentsply Neytech, York, PA, USA) at 270°C for 1 hour, sintered using the Lindberg Furnace (Blue M, Ashville, NC, USA) for 4.5 hours, and cooled down overnight [2]. Ceramic specimens were embedded in acrylic resin using a plastic mold to aid specimen handling during the experiments (**Figure 1A**). Specimens were finished and polished using silicon carbide papers from 240- to 1,200-grit under running water. The embedded Y-TZP specimens were randomly assigned to 3 air-abrasion pressure groups ($n$ = 24): 1 bar (1b), 2 bar (2b), and 2.8 bar (2.8b). The 2.8b group served as the control [25-29]. Each Y-TZP zirconia specimen was air-abraded using airborne-particle abrasion with 50 µm $Al_2O_3$ for 30 seconds (SandStorm Expert, Vaniman Manufacturing Co., Fallbrook, CA, USA) at a 10 mm distance. The surfaces were rinsed with deionized (DI) water for 20 seconds and air-dried for 5 seconds [14].

### Surface roughness measurement

Two representative specimens from each group were scanned prior to the resin cement button preparation via a non-contact 3 dimensional optical profilometer (Proscan 2000, Scantron Industrial Products Ltd., Taunton, UK). Using the S5/03 chromatic sensor, 5 scans/specimen (1 × 1 $mm^2$) were performed to determine surface roughness (step size of 0.01 × 0.01) [2]. All the scanning was completed at a frequency of 300 Hz with full sensor speed (100%). The scans were performed and compared to a non-air-abraded group (control) serving as a reference for the roughness measurements.

### Resin cement button preparation

Each conditioned Y-TZP specimen was placed on an Ultradent jig (Ultradent Products Inc., South Jordan, UT, USA) coupled with a plastic mold that has a cylindrical opening in the middle (2.38 mm in diameter and 3.5 mm in height) to build the resin cement button on Y-TZP (**Figure 1B and 1C**). Panavia F 2.0 (PF) resin cement (Kuraray Noritake Dental Inc., Okayama, Japan) was bonded to the zirconia samples according to the manufacturer's

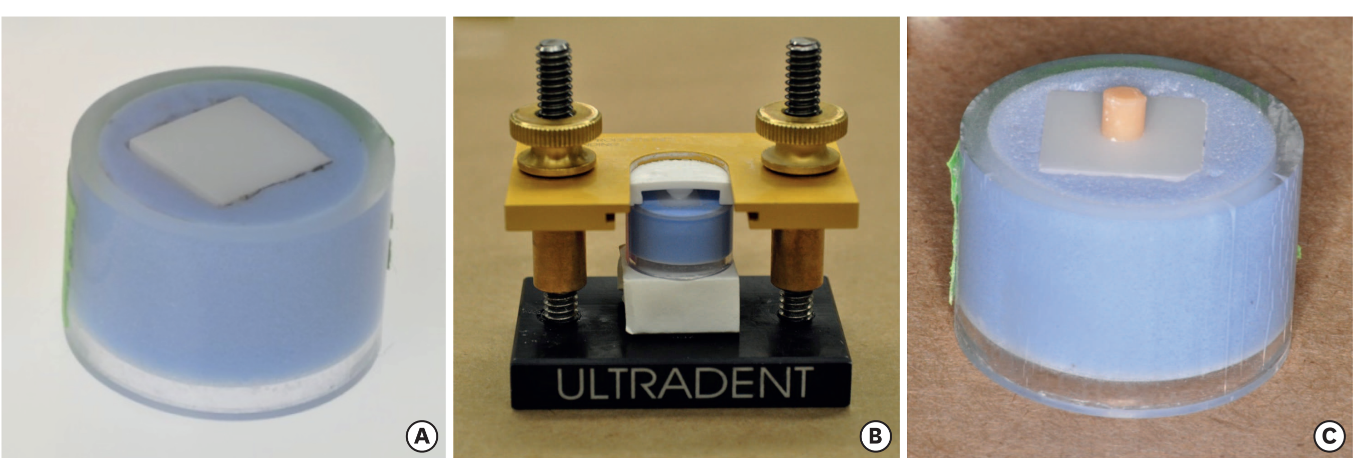


**Figure 1.** Zirconia specimen with resin cement adhered. (A) Zirconia specimen embedded in acrylic resin; (B) Placement of the specimen on the Ultradent jig coupled with the semicircular plastic mold; (C) Zirconia specimen after resin cement button fabrication.


https://doi.org/10.5395/rde.2017.42.3.206

**Table 1.** Material, composition, and application procedure for Panavia F 2.0

| Material | Manufacturer | Lot No. | Composition | Application |
|---|---|---|---|---|
| PF | Kuraray | 061288 | - | - |
| Components | | | | |
| Paste A | - | 00571A | 10-MDP, hydrophobic aromatic and aliphatic photoinitiator, dibenzoyl peroxide dimethacrylate, hydrophilic dimethacrylate, silanized silica | Dispense equal amounts of pastes A and B for 20 sec<br>Apply paste<br>In this study, paste was applied using a syringe and applicator |
| Paste B | - | 00284A | Hydrophobic aromatic and aliphatic dimethacrylate, sodium aromatic sulphinate, N,N-diethanol-p-toluidine, functionalized sodium fluoride, and silanized barium glass | Light cure for 20 sec (LED light) |
| OXYGUARD II | - | 00676A | - | Apply around the margins<br>Wait for 3 min<br>Rinse with distilled water |

10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

instructions (**Table 1**). Briefly, equal amounts of paste A and paste B were mixed on a pad for 20 seconds with a plastic spatula. In order to avoid air bubble entrapment, a syringe and an applicator were used to apply the resin cement into the plastic mold. The specimens were photo-polymerized for 20 seconds using an LED system (DEMI LED, Kerr, Orange, CA, USA). Light irradiance was monitored using a Managing Accurate Light Curing system (MARC, BlueLight Analytics Inc., Halifax, NS, Canada). Light irradiance was approximately 1,000 $mW/cm^2$. OXIGUARD II (Kuraray Noritake Dental Inc.) was then applied around the button and allowed to rest for 3 minutes before being rinsed with DI water. The dimensions of the resin cement buttons were 2.38 mm in diameter and 3.5 mm in height (**Figure 1C**).

### Fatigue CLTC

The specimens prepared in each air-abrasion pressure group were subdivided into 2 groups yielding to 6 groups ($n = 12$). Each prepared specimen with resin cement button was either subjected to SBS testing after 24 hours (1b-24h, 2b-24h, and 2.8b-24h) or to combined fatigue cyclic loading and thermocycling (fatigue cyclic loading, CLTC) (1b-CLTC, 2b-CLTC, and 2.8b-CLTC), and then tested for SBS. Each specimen was loaded on an Ultradent jig, and the designated groups were subjected to fatigue cyclic loading and then tested for SBS. The fatigue cyclic loading and SBS testing was applied in a shear direction parallel to the adhesive interface using an Ultradent semicircular testing fixture (Ultradent Products Inc.) [29]. The semicircular fixture loading area was 2.4 mm in diameter, and was positioned flushed with the Y-TZP specimen surface contacting the cylindrical bonded resin cement at the zirconia and cement interface (**Figure 2**). The fatigue cyclic loading was subjected to a low load (10 N, approximately 2.25 MPa) to prevent loading damage at the zirconia-resin interface for 10,000 cycles and a frequency of 1.0 Hz using a mechanical cycling machine (ElectroPuls E3000, Instron, Norwood, MA, USA) [29]. After completion of the fatigue cyclic loading, the same groups were thermocycled for 5,000 cycles between 6°C–48°C (30 seconds dwell time and 10 seconds transfer time). All groups were then stored in DI water and tested for SBS either after 24 hours or after CLTC.

### SBS test and failure mode analysis

Each specimen was mounted on the Ultradent jig as described earlier and subjected to debonding under shear force using a notched (semicircular) edge at a crosshead speed of 1.0 mm/min (ElectroPuls E3000, Instron; **Figure 2**). The SBS was calculated through the following formula:

$$\text{SBS (MPa)} = \text{Load (N)/area (mm}^2\text{)}$$


https://doi.org/10.5395/rde.2017.42.3.206

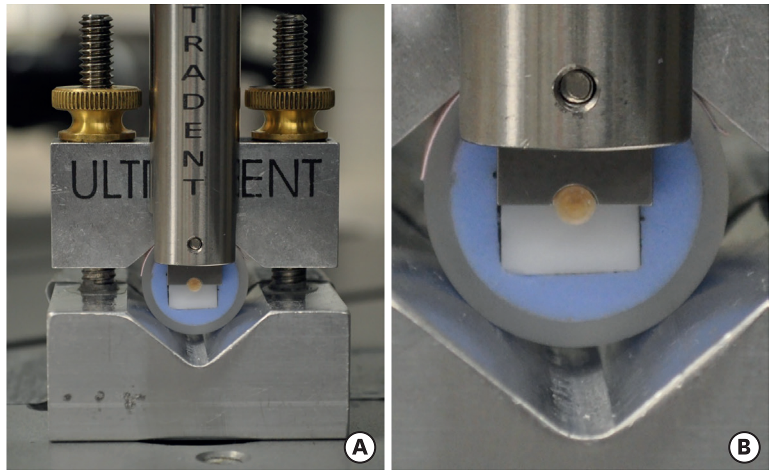


**Figure 2.** Fatigue cyclic loading and shear bond strength test apparatus. Fatigue cyclic loading was applied in a shear direction parallel to the adhesive interface using an Ultradent loading jig with a semicircular loading surface (2.4 mm in diameter) in close proximity to the zirconia-resin button interface and subjected to 10 N load for 10,000 cycles with a frequency of 1.0 Hz. (A) Frontal-view of the testing apparatus; (B) A close up for the testing setup.

Modes of failure were observed with an optical microscope (Measurescope UM-2, Nikon Corporation, Tokyo, Japan) at a magnification of × 40 after SBS testing. The modes of failure were classified as follows: adhesive, failure between the Y-TZP ceramic surface and the resin cement; cohesive, failure within the resin cement; mixed, failures in which partly adhesive and partly cohesive ones were observed coincidently in a fractured surface. Before the resin cement preparation, the surface of representative Y-TZP samples from the control and each air-abrasion treated specimen were prepared to qualitatively analyze the surface roughness under scanning electron microscopy (SEM; JSM 6390 LV, JEOL Ltd., Tokyo, Japan). In addition, SEM images were obtained from fractured/debonded representative specimens. Specimens were sputter-coated with gold for 90 seconds (Desk II Cold Sputter, Denton Vacuum LLC, Moorestown, NJ, USA) prior to SEM imaging.

### Statistical analysis

Comparisons between groups for SBS values were performed using 2-way analysis of variance (ANOVA), followed by *post-hoc* comparisons. Specimens with pre-test failures were included in the analysis as 0 MPa; the lowest observed value was 1.8 MPa in group 1b-24h. Weibull characteristic strengths were compared using parametric Weibull-model survival analysis. Weibull moduli and their corresponding standard errors were estimated for each group using the survival analysis, and compared pair-wise between groups using z-tests. The differences between the groups for type of failure were analyzed using Fisher's exact tests. ANOVA was performed to compare the surface roughness between groups, with a fixed effect for the groups and a random effect to account for correlations among multiple roughness measurements within one specimen.

## RESULTS

### Surface roughness measurement

The mean average surface roughness (Ra) is shown in **Figure 3**. The 2.8b group showed significantly higher Ra than that of the control group and 1b (Ra, $p = 0.006$, $p = 0.017$, respectively). No other statistically significant differences were found between other groups.

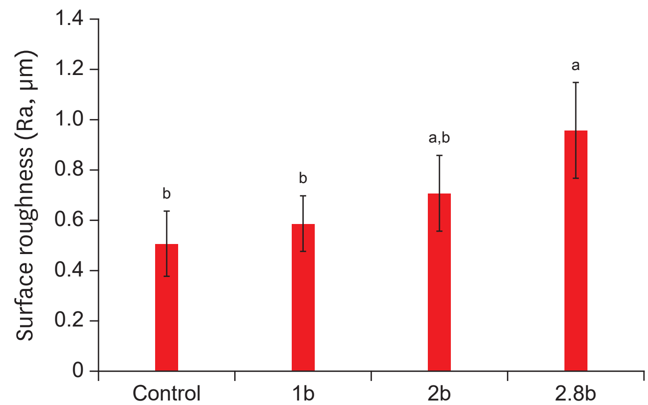


**Figure 3.** Mean surface roughness and standard deviations of different groups after air-abrasion. Control group represents the zirconia surface before air ab-rasion treatment.
Ra, average surface roughness; 1b, 1 bar; 2b, 2 bar; 2.8b, 2.8 bar.
[a,b]Different letters represent significant differences among the air-abrasion pressures tested.

### SBS test and failure mode analysis

SBS data indicated that the interaction effect between pressure and time/cycling was not significant ($p$ = 0.220, **Table 2**). Additionally, pressure did not have a significant effect on SBS. Mean SBS ($p$ = 0.006) and Weibull characteristic strength ($p$ = 0.012) were significantly higher for the 24-hour storage groups compared to the CLTC groups. Also, the 2b-CLTC group had significantly lower Weibull modulus than those of the other groups ($p < 0.05$).

The 2b-CLTC group presented significantly higher percentage of specimens failing during fatigue test than those of the 1b-24h ($p$ = 0.037), 2b-24h ($p$ = 0.042), 2.8b-24h ($p$ = 0.042), and 2.8b-CLTC ($p$ = 0.042) groups. None of the other groups showed significantly different failure modes from each other (**Table 3**). In general, the mixed failure mode was observed more than

**Table 2.** Mean and SD of the shear bond strength (in MPa)

| Group | SBS | Weibull characteristic strength | Weibull modulus |
|---|---|---|---|
| 1b-24h | 9.2 ± 3.4[a] | 10.2 (8.3–12.2)[a] | 3.1 (1.7–4.6)[a] |
| 2b-24h | 10.5 ± 3.0[a] | 11.6 (9.9–13.4)[a] | 4.0 (2.3–5.7)[a] |
| 2.8b-24h | 10.7 ± 5.9[a] | 12.1 (8.5–15.7)[a] | 2.0 (1.2–2.9)[a] |
| 1b-CLTC | 8.7 ± 4.2[b] | 9.4 (6.4–12.5)[b] | 1.8 (0.9–2.7)[a] |
| 2b-CLTC | 5.8 ± 5.3[b] | 4.3 (0.0–8.6)[b] | 0.6 (0.3–0.9)[b] |
| 2.8b-CLTC | 7.6 ± 1.9[b] | 8.3 (7.2–9.4)[b] | 4.5 (2.5–6.4)[a] |

Values are presented as mean ± SD or number (95% CI). Mean SBS was significantly higher ($p$ = 0.006) along with Weibull characteristic strength ($p$ = 0.012) for 24-hour storage compared to CLTC. 2b-CLTC had significantly lower Weibull modulus than the other groups ($p < 0.05$).
SD, standard deviation; SBS, shear bond strength; CI, confidence interval; 1b, 1 bar; 2b, 2 bar; 2.8b, 2.8 bar; 24h, 24 hours; CLTC, fatigue cyclic loading and thermocycling.
[a,b]Superscript lowercase letters represent significant differences within the same column.

**Table 3.** Failure mode of the samples

| Group | Adhesive failure | | Mixed failure | | Failed during cyclic loading | |
|---|---|---|---|---|---|---|
| | No. | % | No. | % | No. | % |
| 1b-24h[b] | 1 | 8 | 11 | 92 | 0 | 0 |
| 2b-24h[b] | 3 | 25 | 9 | 75 | 0 | 0 |
| 2.8b-24h[b] | 4 | 33 | 8 | 67 | 0 | 0 |
| 1b-CLTC[a,b] | 0 | 0 | 11 | 92 | 1 | 8 |
| 2b-CLTC[a] | 1 | 8 | 6 | 50 | 5 | 42 |
| 2.8b-CLTC[b] | 4 | 33 | 8 | 67 | 0 | 0 |

The description of failure modes is follow as; adhesive failure, failure at the interface between Y-TZP zirconia and resin cement; cohesive failure, failure within the resin cement; and mixed failure, failure including both adhesive and cohesive failure.
Y-TZP, yttrium oxide-stabilized tetragonal zirconia polycrystal; 1b, 1 bar; 2b, 2 bar; 2.8b, 2.8 bar; 24h, 24 hours; CLTC, fatigue cyclic loading and thermocycling.
[a,b]Superscript lowercase letters represent significant differences among the groups.

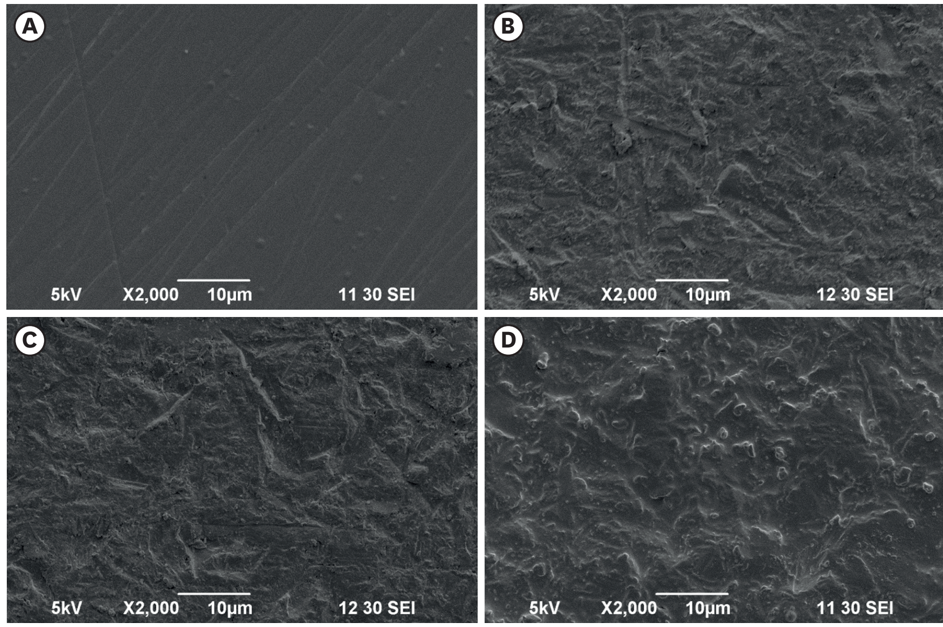


**Figure 4.** Scanning electron microscopic images (× 2,000) of zirconia surface for control and after different air-abrasion pressures. (A) Control group (no air-abrasion); (B) 1 bar; (C) 2 bar; (D) 2.8 bar.

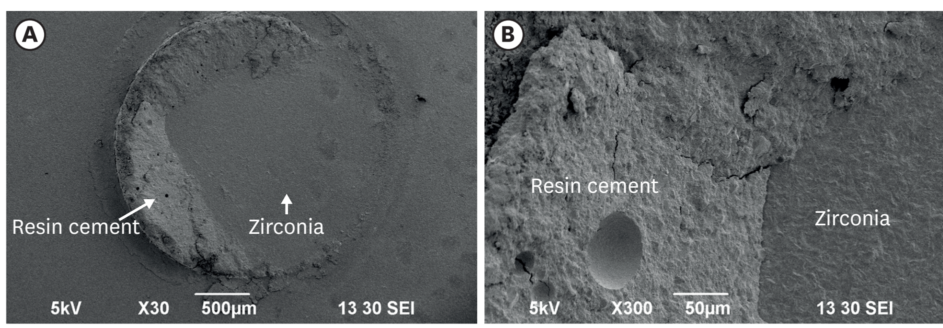


**Figure 5.** Scanning electron microscopic images of zirconia surface denoting mixed mode of failure after debonding at magnification (A) × 30; (B) × 300.

the adhesive one. Representative SEM images of specimens air-abraded at different pressures demonstrated a qualitative increase in surface roughness with increasing pressure (**Figure 4**). **Figure 5** shows SEM images of a representative specimen with mixed failure.

## DISCUSSION

The main goal of the present study was to investigate whether fatigue CLTC could serve as an aging method to evaluate resin bond durability to zirconia. According to the present findings, the null hypothesis was rejected as the combined aging method significantly decreased resin bond strength to zirconia regardless of the air-abrasion pressure used to condition the ceramic surface.

In this study, the fatigue cyclic loading was performed on the bond interface of adhesive cement and zirconia in a shear direction. This methodology was devised in an attempt to allow the application of cyclic low loads to the bonded interface, which may better mimic the


https://doi.org/10.5395/rde.2017.42.3.206

fatigue environment occurring in the oral cavity [24,29]. The results of this study show that interfacial adhesion of MDP-containing cement—zirconia is indeed susceptible to mechanical degradation, although the optimal stress for use in the method needs further investigation.

In the present investigation, the zirconia surface roughness data obtained using non-contact profilometry (quantitative) and SEM images (qualitative) after air-abrasion confirm the increase in surface irregularities with increasing air-abrasion pressure. Although the surface roughness for group 2b was not significantly different from those of 1b and 2.8b, there was a significant increase in surface roughness between 1b and 2.8b. These observations are in agreement with the results of a recent study in which the SEM images showed differences between 1b and 2.8b [25].

It is well established that the luting agent plays a critical role in the long-term success of resin-ceramic bonding. The resin cement used (PF) contained a phosphate monomer (10-MDP). The phosphate ester monomer in 10-MDP is suggested to enhance bond strength due to the chemical P-O-Zr bond formed between zirconia and MDP [8,11,14-21,30]. Furthermore, the bond strength between the MDP-containing resin cement and zirconia is suggested to be enhanced when the zirconia surface is air-abraded with alumina particles, therefore, a 2-fold bonding is produced, namely chemical bonding and micromechanical interlocking [30-32]. In this study, PF showed significantly lower SBS after the combined CLTC processes. The significantly higher SBS observed for 24-hour storage compared to CLTC suggests that CLTC had significant effect on the strength of bonded cement to zirconia. Worth mentioning, only 2b-CLTC group showed a large number of specimens failing during fatigue-cyclic loading and before shear bond testing. This behavior resulted in the significantly lower Weibull modulus for 2b-CLTC.

According to Nemli *et al.* [33], cyclic fatigue can cause phase transformation of tetragonal crystals of Y-TZP to monoclinic crystal structures. Therefore, one would expect that cyclic fatigue would lead to some degree of phase transformation. However, the low stress value used (~2.25 MPa) may not have resulted in significant transformation. Meanwhile, the association between the surface roughness and resin cement is particularly critical when using low air-abrasion pressure as the topography created may not be deep enough to properly impregnate the resin cement in the micro-irregularities on the zirconia surface [19-21]. However, this was not supported in this study, where group 1b may have generated sufficient roughness and surface morphology for satisfactory bonding to zirconia compared to group 2b.

The proposed testing methodology may give a more relevant evaluation of bond strength between zirconia and adhesive cements as it better represents the worst case in the oral cavity. Further investigations are needed to validate this testing method by addressing the effect of increasing the load and fatigue cycle number, testing different adhesive cements and surface treatments. Additional research of the phase transformation and surface flaw geometry that occurs during specimen preparation is also worth including in future studies.

## CONCLUSIONS

Based on the results of this study, 2.8b air-abrasion resulted in higher surface roughness and increased the SBS of resin cement to zirconia. The combined CLTC significantly decreased the SBS of resin cement to zirconia regardless of the air-abrasion pressure used to condition the zirconia surfaces.


https://doi.org/10.5395/rde.2017.42.3.206

## REFERENCES


1. Lazar DR, Bottino MC, Özcan M, Valandro LF, Amaral R, Ussui V, Bressiani AH. Y-TZP ceramic processing from coprecipitated powders: a comparative study with three commercial dental ceramics. Dent Mater 2008;24:1676-1685.
PUBMED | CROSSREF
2. Luangruangrong P, Cook NB, Sabrah AH, Hara AT, Bottino MC. Influence of full-contour zirconia surface roughness on wear of glass-ceramics. J Prosthodont 2014;23:198-205.
PUBMED | CROSSREF
3. Denry I, Kelly JR. Emerging ceramic-based materials for dentistry. J Dent Res 2014;93:1235-1242.
PUBMED | CROSSREF
4. Lawson NC, Burgess JO. Dental ceramics: a current review. Compend Contin Educ Dent 2014;35:161-166.
PUBMED
5. Wang H, Aboushelib MN, Feilzer AJ. Strength influencing variables on CAD/CAM zirconia frameworks. Dent Mater 2008;24:633-638.
PUBMED | CROSSREF
6. Lohbauer U, Zipperle M, Rischka K, Petschelt A, Müller FA. Hydroxylation of dental zirconia surfaces: characterization and bonding potential. J Biomed Mater Res B Appl Biomater 2008;87:461-467.
PUBMED | CROSSREF
7. Wegner SM, Kern M. Long-term resin bond strength to zirconia ceramic. J Adhes Dent 2000;2:139-147.
PUBMED
8. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. J Prosthet Dent 2003;89:268-274.
PUBMED | CROSSREF
9. Hummel M, Kern M. Durability of the resin bond strength to the alumina ceramic Procera. Dent Mater 2004;20:498-508.
PUBMED | CROSSREF
10. Friederich R, Kern M. Resin bond strength to densely sintered alumina ceramic. Int J Prosthodont 2002;15:333-338.
PUBMED
11. Kern M. Bonding to oxide ceramics—laboratory testing versus clinical outcome. Dent Mater 2015;31:8-14.
PUBMED | CROSSREF
12. Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/cementation to zirconia and other non-silicate ceramics: where are we now? Dent Mater 2011;27:71-82.
PUBMED | CROSSREF
13. Seo DG. Zirconia surface treatment for successful bonding. Restor Dent Endod 2014;39:333.
PUBMED | CROSSREF
14. Bottino MA, Bergoli C, Lima EG, Marocho SM, Souza RO, Valandro LF. Bonding of Y-TZP to dentin: effects of Y-TZP surface conditioning, resin cement type, and aging. Oper Dent 2014;39:291-300.
PUBMED | CROSSREF
15. Inokoshi M, De Munck J, Minakuchi S, Van Meerbeek B. Meta-analysis of bonding effectiveness to zirconia ceramics. J Dent Res 2014;93:329-334.
PUBMED | CROSSREF
16. de Souza G, Hennig D, Aggarwal A, Tam LE. The use of MDP-based materials for bonding to zirconia. J Prosthet Dent 2014;112:895-902.
PUBMED | CROSSREF
17. Blatz MB, Sadan A, Martin J, Lang B. *In vitro* evaluation of shear bond strengths of resin to densely-sintered high-purity zirconium-oxide ceramic after long-term storage and thermal cycling. J Prosthet Dent 2004;91:356-362.
PUBMED | CROSSREF
18. Özcan M, Bernasconi M. Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis. J Adhes Dent 2015;17:7-26.
PUBMED
19. Özcan M, Melo RM, Souza RO, Machado JP, Felipe Valandro L, Botttino MA. Effect of air-particle abrasion protocols on the biaxial flexural strength, surface characteristics and phase transformation of zirconia after cyclic loading. J Mech Behav Biomed Mater 2013;20:19-28.
PUBMED | CROSSREF
20. Kern M, Barloi A, Yang B. Surface conditioning influences zirconia ceramic bonding. J Dent Res 2009;88:817-822.
PUBMED | CROSSREF

21. Aurélio IL, Marchionatti AM, Montagner AF, May LG, Soares FZ. Does air particle abrasion affect the flexural strength and phase transformation of Y-TZP? A systematic review and meta-analysis. Dent Mater 2016;32:827-845.
PUBMED | CROSSREF

22. Barkmeier WW, Erickson RL, Latta MA. Fatigue limits of enamel bonds with moist and dry techniques. Dent Mater 2009;25:1527-1531.
PUBMED | CROSSREF

23. Wegner SM, Gerdes W, Kern M. Effect of different artificial aging conditions on ceramic-composite bond strength. Int J Prosthodont 2002;15:267-272.
PUBMED

24. Williamson RT, Mitchell RJ, Breeding LC. The effect of fatigue on the shear bond strength of resin bonded to porcelain. J Prosthodont 1993;2:115-119.
PUBMED | CROSSREF

25. Re D, Augusti D, Augusti G, Giovannetti A. Early bond strength to low-pressure sandblasted zirconia: evaluation of a self-adhesive cement. Eur J Esthet Dent 2012;7:164-175.
PUBMED

26. Zhang Y, Lawn BR, Malament KA, Van Thompson P, Rekow ED. Damage accumulation and fatigue life of particle-abraded ceramics. Int J Prosthodont 2006;19:442-448.
PUBMED

27. Zhang Y, Lawn BR, Rekow ED, Thompson VP. Effect of sandblasting on the long-term performance of dental ceramics. J Biomed Mater Res B Appl Biomater 2004;71:381-386.
PUBMED | CROSSREF

28. Özcan M, Kerkdijk S, Valandro LF. Comparison of resin cement adhesion to Y-TZP ceramic following manufacturers' instructions of the cements only. Clin Oral Investig 2008;12:279-282.
PUBMED | CROSSREF

29. Nishigori A, Yoshida T, Bottino MC, Platt JA. Influence of zirconia surface treatment on veneering porcelain shear bond strength after cyclic loading. J Prosthet Dent 2014;112:1392-1398.
PUBMED | CROSSREF

30. Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. Dent Mater 1998;14:64-71.
PUBMED | CROSSREF

31. Chang JC, Powers JM, Hart D. Bond strength of composite to alloy treated with bonding systems. J Prosthodont 1993;2:110-114.
PUBMED | CROSSREF

32. Chen L, Suh BI, Brown D, Chen X. Bonding of primed zirconia ceramics: evidence of chemical bonding and improved bond strengths. Am J Dent 2012;25:103-108.
PUBMED

33. Nemli SK, Yilmaz H, Aydin C, Bal BT, Tıraş T. Effect of fatigue on fracture toughness and phase transformation of Y-TZP ceramics by X-ray diffraction and Raman spectroscopy. J Biomed Mater Res B Appl Biomater 2012;100:416-424.
PUBMED | CROSSREF